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(54) **Thermal treatment of brazed products for improved corrosion resistance.**

(57) Corrosion-resistance in a brazed aluminium product is improved by subjecting the product to a heat treatment at a temperature of 300° to 800°F (150° to 425°C) for at least 25 minutes, preferably preceded by cooling the product to a temperature below the temperature ultimately selected for the treatment and in any event below about 550°F (290°C).

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THERMAL TREATMENT OF BRAZED PRODUCTS FOR IMPROVED CORROSION RESISTANCE

BACKGROUND OF THE INVENTION

This invention relates to brazed aluminum products, and particularly to the behavior of such products when exposed to corrosive environments.

Brazing is a widely used process for joining aluminum parts in the manufacture of such products as air conditioning evaporators and radiators. In use, these products, particularly automotive radiators, are exposed to salted road splash causing intergranular corrosion attack, which limits the useful life of the product.

This problem has been addressed in the literature in a variety of ways. An early example is Miller, U.S. Patent No. 2,821,014 (January 28, 1958), where it is disclosed that intergranular corrosion problems in flux and dip brazing are alleviated by adding an interlayer between the structural member portion and the brazing layer. The interlayer is aluminum or an aluminum-base alloy, particularly certain magnesium-containing alloys, having a melting point greater than that of the structural alloy. The solution offered by Singleton et al., U.S. Patent No. 3,788,824 (January 29, 1974) and its divisional, No. 3,881,879 (May 6, 1975), is directed to vacuum brazing, and involves the addition of iron to either the core alloy or the cladding alloy as an alloying element, resulting in improvements in both corrosion resistance and sag resistance. Anthony et al., U.S. Patent No. 4,039,298 (August 2, 1977) address both flux and vacuum brazing, and disclose a composite of complex and highly specified composition as being particularly beneficial in terms of corrosion properties. The disclosed core alloy contains specified amounts of manganese, copper, chromium, silicon and iron as alloying elements with both a solid solution and an alpha-phase, whereas the alloying elements in the cladding are bismuth and silicon. An additional disclosure by the same patentees appears in U.S. Patent No. 4,093,782 (June 6, 1978) and its continuation-in-part, No. 4,167,410 (September 11, 1979), in which the core alloy contains a specified combination of chromium and manganese, with resultant improvements in both corrosion resistance and sag resistance. A similar disclosure appears in Setzer et al., U.S. Patent No. 3,994,695 (November 30, 1976), where the core alloy contains a chromium-manganese-zirconium combination, the sole claimed benefit however being an improvement in sag resistance. A combination of copper and titanium as primary alloying elements in the core alloy is disclosed in Kaifu et al., U.S. Patent No. 4,339,510 (July 13, 1982), as providing a benefit in intergranular corrosion resistance.

A different approach is disclosed by Nakamura, U.S. Patent No. 4,172,548 (October 30, 1979), in which corrosion following fluxless brazing processes in general (including both vacuum brazing and brazing in an inert atmosphere) is controlled by controlling the grain size of the brazing sheet to at least 60 microns in diameter, achieved by a controlled cold work followed by a full anneal.

Heat treatment is disclosed for metallic alloys in general for a number of reasons. Heat treatment after brazing in a heat-hardenable copper-based alloy is disclosed by Silliman, U.S. Patent No. 2,117,106 (May 10, 1938) for returning hardness and spring qualities lost during the brazing procedure. Soldered joints in copper-brass radiators are heat-treated in a process disclosed by Harvey, U.S. Patent No. 3,335,284 (November 28, 1967) to lessen the occurrence of stress and creep-rupture at the operating temperature. Tisinai, et al., U.S. Patent No. 3,028,268 (April 3, 1962) used a high temperature heat treatment to impart corrosion resistance to nickel-chromium-molybdenum alloys. Heat treatments are disclosed for similar purposes in aluminum wires (Weber, U.S. Patent No. 3,503,596, March 31, 1970) and zinc-based alloys (Gervais, et al., U.S. Patent No. 3,880,679, April 29, 1975).

SUMMARY OF THE INVENTION

It has now been discovered that the heightened corrosion susceptibility in aluminum alloy products brought about by brazing processes can be reduced by a post-brazing heat treatment at a temperature below that which causes solution heat treatm nt. The treatment has the capacity of increasing the useful life of a brazed product several-fold, and is particularly unusual and unexpected when applied to brazed products made from a non-heat-treatable core material.

DETAILED DESCRIPTION OF THE INVENTION AND PREFERRED EMBODIMENTS

The duration and intensity of the heat treatment in accordance with the present invention are interdependent in a reciprocal manner. Thus, as higher temperatures are used, equivalent results may be obtained in lesser times, and vice versa. With these considerations in mind, the parameters of the treatment may be varied widely. In general, however, the temperature is kept below that at which solution heat treatment is known to occur for the particular core alloy used in the product. Best results will generally occur at temperatures ranging from about 300°F (150°C) to about 800°F (430°C), preferably from about 450°F (230°C) to about 650°F (345°C).

In the broadest sense, the length of time during which the product is held at the treatment temperature may range from a fraction of an hour up to several days. The time requirement for any particular desired result will vary with the treatment temperature. At treatment temperatures less than about 400°F (204°C), best results will be obtained in treatments lasting at least about 20 hours; while at temperatures of about 400°F (204°C) to about 500°F (260°C), the time period for comparable results is at least about 10 hours; and above about 500°F (260°C), the time period may be as low as about 25 minutes. In preferred embodiments of the invention, the treatment is done at a temperature of about 450°F (230°C) to about 650°F (345°C) for at least about two hours, with commercially useful results generally occurring in less than ten hours. The pressure maintained during the treatment may vary widely; atmospheric pressure is sufficient.

Since the heat treatment is well below the brazing temperature range, cooling will be required between the two procedures. Beneficial results will generally be obtained, regardless of whether the cooling is done quickly or gradually. The preferred method, however, is to cool the brazed product to a temperature w ll below the heat treatment temperature, then raise the temperature back to the desired level. Beneficial effects will thus result by lowering the temperature to about 550°F (288°C) or less, preferably about 350°F (177°C) or less, in this intermediate stage.

The process of the invention is applicable to brazed products in general, with particular utility in connection with vacuum-brazed products. The process is preferably applied to products formed from non-heat-treatable core alloys, preferably those of the 3XXX series of alloys as designated by the Aluminum Association. Examples are the 3003, 3005, and 3105 alloys, including modified versions of these alloys. Products of particular interest are also those having cladding or brazing alloys which contain silicon as the primary alloying element. Exemplary such alloys are those of the 4XXX series, notably the 4003, 4043, 4045, 4047, 4104 and 4343 alloys, including modified versions of these alloys.

The following examples are offered for purposes of illustration only, and are intended neither to define nor limit the invention in any manner.

EXAMPLE 1

A brazing sheet of the following composition was cut into pieces measuring 2-3/16 in. (5.56 cm) by 4-3/4 in. (12.06 cm) by 0.0206 in. (0.052 cm) thick:

Table 1.1
TEST MATERIALS

Element	Core Alloy Weight %	Cladding Alloy (12% Both Sides) Weight %
Si	0.14	11.7
Fe	0.49	0.3
Cu	0.10	0.05
Mn	1.12	0.01
Mg	0.56	0.19
Cr	0.007	0.002
Zn	0.07	0.03
Ti	0.019	0.027
Al	balance	balance

A simulated brazing procedure was performed by suspending the pieces upright in a vacuum furnace where they were heated to 1100°F (593°C) at a pressure of 1×10^{-5} torr in fifteen minutes and held at temperature for two minutes. The pieces were then withdrawn from the furnace and cooled in air. Some of the pieces were then given a post-braze thermal treatment in accordance with the present invention by being heated to 350°F (177°C) at atmospheric pressure for varying lengths of time.

All pieces were then cut to fit into a leak test device, and one side of each was masked with electroplater's tape. The pieces were then corrosion tested in the device according to ASTM Procedure No. G43 in a 98% humid atmosphere by intermittent exposure to an atomized solution consisting of (on weight basis):

Table 1.2
CORROSION SOLUTION

Component	Amount
Synthetic sea salt	42 g/liter
Glacial acetic acid	10 ml/liter
Water	balance

for extended periods of time. This is a procedure which is known in the aluminum industry for its ability to duplicate the intergranular mode of corrosion observed in automotive radiators retrieved from field service.

During exposure to the corrosion solution, the pieces were monitored daily for the development of blisters on the taped sid indicating perforation of the pieces by corrosion. The results are shown in Table 1.3.

Table 1.3

BLISTER TEST RESULTS

Post-braze Treatment (Hours at 350°F)	Hours Exposure to Corrosion Solution	Appearance of Taped Side
4	48	blisters
8	48	blisters
24	48	no blisters
24	160	blisters
74	160	no blisters
74	600	blisters

Summarizing this table, corrosion resistance improved as the post-braze treatment was extended from 4 to 24 hours (based on a 48-hour corrosion test), and again from 24 to 74 hours (based on a 160-hour corrosion test). The point where blisters (i.e., perforation due to corrosion) did appear on a sample which had been given a 74-hour post-braze treatment at 350°F was between 160 and 600 hours' exposure to the corrosion solution.

The tape was then removed from the test pieces, and the pieces were rinsed and cleaned in a solution of chromic and phosphoric acids (ASTM Solution No. G1) at 180°F (82°C), then baked for thirty minutes at 250°F (121°C) to remove retained water.

Each piece was then pressurized with air at 20 psig (13.8 newtons/cm²) under water and the number of perforations detected by visual observation of streams of bubbles. The number of perforations per piece is shown in Table 1.4 as a function of the degree of post-braze treatment and the degree of exposure to th corrosion solution:

Table 1.4

PERFORATION TEST RESULTS

Post-braze Treatment (Hours at 350°F)	Hours Exposure to Corrosion Solution	Number of Perforations
0	48	15+*
4	48	25+*
8	48	10
24	48	0
24	160	7
74	160	0
74	600	2

* Where the perforations were either so numerous or so large as to make counting uncertain, an estimate was made and a "+" sign placed next to the number.

Summarizing Table 1.4, post-braze treatments of 8 hours and above produced consistent reductions of the number of leaks within any given number of hours of corrosion exposure.

EXAMPLE 2

The procedures of Example 1 w re repeated, using the same materials, with the xception that the temperature in the post-braze thermal treatment was varied between 250°F (121°C) and 550°F (288°C). The results are shown in Table 2.1.

Table 2.1

BLISTER AND PERFORATION TEST RESULTS

Post-Braze Treatment Temperature	Time	Hours to First Blister	Total Hours Of Exposure	Number Of Perforations
-	0	48	48	15+
250°F	24 h	24	48	19+
	72 h	24	48	10+
	100 days	144	144	2
350°F	24 h	144	168	2
	72 h	216	264	1
450°F	0.5 h	48	48	20+
	1.5 h	24	48	17+
	24 h	216	264	2
	72 h	240	264	1
500°F	0.1 h	48	48	15+
	0.3 h	48	48	14+
	24 h	216*	264	2
	72 h	---	1108	0
550°F	1 min	24	48	9+
	6 min	48*	48	4+
	24 h	--*	1108	3
	72 h	--	1108	2

* No blisters observed over entire duration of test.

Summarizing Table 2.1, there is essentially no improvement obtained from post-braze treatments of 24 and 72 hours at 250°F when compared to the untreated (i.e., as-brazed) sample which is the first entry. Significant effects are seen at higher temperatures, however. For example, a 24-hour treatment at 350°F was equal in effectiveness to a 100-day treatment at 250°F. Among the 350°F tests, an extension of th treatment time from 24 to 72 hours produced a further improvement, while equivalent improvements were achieved in 24 hours at 450°F and 500°F. Further extension to 72 hours at 500°F produced an even larger improvement, which was obtained in only 24 hours at 550°F. Thus, over the temperature range investigated, equivalent improvements occur in shorter times at higher temperatures.

EXAMPLE 3

Two vacuum brazed automotive radiators formed from the following core and brazing alloys w re selected for testing:

Table 3.1

RADIATOR MATERIALS

	Radiator A		Radiator B	
Element	Core	Cladding	Core	Cladding
Si	.21	9.6	.14	11.7
Fe	.55	.4	.49	.3
Cu	.15	.16	.10	.05
Mn	1.18	.08	1.12	.01
Mg	.01	1.45	.56	.19
Zn	.08	.15	.07	.02
Ti	.02	--	.01	.03
Al	balance	balance	balance	balance

Test sections were cut from each radiator, each about 6 inches (15.2 cm) long and containing four tubes with intervening air centers. Reserving one section of each type as controls, the remaining sections were subjected to a post-braze heat treatment at 350°F (177°C) at atmospheric pressure for 88 hours.

The ends of the tubes were sealed off for the corrosion test, which consisted of exposing the sampl s to a corrosion solution as in Example 1 for preselected periods of time. After exposure of the samples, the plugs were removed and the samples were cleaned and dried as in Example 1. The two interior tubes in each sample were submerged and pressurized with air at 20 psig and perforation observations were mad as before. The results are shown in Table 3.2.

Table 3.2

PERFORATION TEST RESULTS

	Hours Exposure to Corrosion Solution	Number of Perforations Tube 1	Number of Perforations Tube 2
Radiator A			
As Brazed	144	25+	25+
	264	12+	12+
Thermally Treated 350°F/88 hrs	144	0	0
	264	0	0
	408	2	3
	600	5	4
Radiator B			
As Brazed	264	25+	25+
Thermally Treated 350°F/88 hrs	264	0	0
	408	1	0
	600	0	0
	1008	2	0

Summarizing Table 3.2, major improvements in corrosion resistance are shown in all thermally treated samples, even with four times the exposure to the corrosion solution.

EXAMPLE 4

The procedures of Example 1 were again repeated, using the same materials but at temperatures of 550°F (288°C) and 650°F (343°C) while varying the treatment time further. The results are shown in Table 4.1, which presents data from two samples for each set of conditions.

Table 4.1

BLISTER AND PERFORATION TEST RESULTS

Post-Braze Treatment Temperature	Post-Braze Treatment Time	Hours to First Blister	Total Hours Of Exposure	Number Of Perforations
550°F	24 h	-	1008	0
		-	1008	0
550°F	16 h	-	1008	0
		-	1008	0
550°F	8 h	-	1008	0
		-	1008	0
550°F	4 h	-	1008	0
		-	1008	0
550°F	2 h	336	336	1
		-	1008	0
550°F	1 h	168	168	4
		216	216	1
550°F	0.5 h	168	168	2
		192	192	2
650°F	1 h	528	528	2
		504	504	2

Summarizing this table and comparing to the first entry in Table 2.1, improvements in corrosion resistance occur with treatments at 550°F lasting as little as 0.5 hour, although further improvement occurs with longer treatment times.

EXAMPLE 5

The procedures of Example 1 were again repeated with the same materials, this time raising th treatment temperature even further to show a loss of improvement at high temperatures. The results are listed in Table 5.1, again showing data from two samples for each set of conditions.

Table 5.1

BLISTER AND PERFORATION TEST RESULTS

Post-Braze Treatment Temperature	Time	Hours to First Blister	Total Hours Of Exposure	Number Of Perforations
650°F	25 h	240	288	2
		456	456	1
	50 h	672	672	2
		720	792	4
	75 h	312	336	1
		648	672	1
700°F	25 h	288	288	0
		432	456	1
	50 h	384	384	1
		336	336	1
	75 h	356	384	3
		288	288	1
750°F	25 h	288	288	3
		312	336	3
	50 h	336	336	1
		288	288	1
	75 h	216	216	3
		264	288	3
800°F	25 h	192	192	4
		192	192	3
	50 h	216	216	5
		144	144	1
	75 h	120	120	1
		216	216	8
850°F	25 h	96	120	20
		48	120	20
	50 h	48	48	20
		72	120	20
	75 h	48	48	20
		48	120	20
900°F	25 h	48	48	20
		96	120	20

	50 h	48	48	20
		72	120	20
1000°F	25 h	48	48	0
		120	144	10
	50 h	120	144	15
		120	288	0
1050°F	25 h	120	216	6
		120	216	12
	50 h	120	144	15
		192	288	0
		144	144	20
		288	288	0

Summarizing this table and again comparing to the first entry in Table 2.1, a loss of effectiveness is seen at treatment temperatures of 850°F (454°C) and above.

EXAMPLE 6

This example demonstrates the effect achieved by inserting an intermediate cooling step between the brazing and heat treatment steps. In Table 6.1 below, the heat treatment was performed without an intermediate cooling, by merely placing the samples in a chamber at the temperature shown, for the duration shown, immediately after removal from the brazing furnace. The materials and all other procedures were the same as in Example 1.

Table 6.1

POST-BRAZE HEAT TREATMENT WITHOUT INTERMEDIATE COOLING -- BLISTER AND PERFORATION TEST RESULTS

Post-Braze Treatment		Hours to 1st Blister		[Hrs Exp]/[# Perfs]*	
Temperature	Time	Sample: 1	2	1	2
800°F	25 min	48	48	48/20	48/20
	50	72	72	72/4	72/4
750°F	25	48	48	48/20	48/20
	50	48	48	48/4	48/6
700°F	25	48	48	48/20	48/20
	50	48	48	48/10	48/5
	75	48	48	48/9	48/6
650°F	25	48	48	48/20	48/12
	50	72	72	72/20	72/12
	75	48	72	48/2	72/14
600°F	25	48	48	48/10	48/7
500°F	50	48	48	48/20	48/20
400°F	50	48	48	48/20	48/20

* I.e., [Total Hours of Exposure]/[Number of Perforations]

In Table 6.2, the samples were cooled between the brazing and heat treatment steps to the temperatures shown and held there momentarily before being reheated to 650°F (343°C) for the corrosion resistance treatment.

Table 6.2

POST-BRAZE HEAT TREATMENT WITH INTERMEDIATE COOLING -- BLISTER AND PERFORATION TEST RESULTS

Post-Braze Treatment Cool-Down Temp.	Reheat Temp.	Time At Reheat	Hours To 1st Blister	Total Hours Of Exp.	No. Of Perfs.
550°F	650°F	50 h	96	120	3
			96	120	4
400	650	50	96	120	6
			120	120	2
350	650	50	216	216	1
			264	312	3
300	650	50	264	312	4
			216	216	1
250	650	50	288	312	2
			288	312	2

Summarizing Tables 6.1 and 6.2, the intermediate cool-down to a temperature of 550°F or lower followed by reheating to a higher temperature provides a significant improvement in the ultimate corrosion resistance, the improvement increasing as the cool-down temperature is lowered.

The foregoing is intended primarily for purposes of illustration. It will be readily apparent to those skilled in the art that numerous modifications and variations of the procedures and conditions described above may be made without departing from the spirit and scope of the invention.

Claims

1. A method for improving corrosion-resistance in a brazed aluminium-base product, characterised by heating the product to a temperature in the range from 300° to 800°F (150° to 425°C) for at least 25 minutes.

2. A method in accordance with claim 1, in which the temperature is in the range from 450° to 650°F (233° to 345°C).

3. A method in accordance with claim 1 or 2, in which the heating is continued for at least 2 hours.

4. A method in accordance with any preceding claim, in which the product is cooled after brazing to an intermediate temperature below the heating temperature and less than about 550°F (290°C) prior to the heating.

5. A method in accordance with claim 4, in which the product after brazing is cooled to an intermediate temperature below the heating temperature and less than about 350°F (175°C) prior to the heating.

6. A method in accordance with any preceding claim, in which the product is heated to a temperature below that at which solution heat treatment is caused.

7. A method for producing a brazed aluminium product having improved resistance to intergranular corrosion,
characterised by

(a) joining a first aluminium-base material comprising an aluminium-base core material clad with an aluminium-base brazing alloy to a second aluminium-base material by brazing; and

(b) heating the product of step (a) to a temperature in the range from 300° to 800°F (150° to 425°C) for at least 25 minutes.

8. A method in accordance with claim 7, in which the aluminium-base core material is a non-heat-treatable aluminium-base alloy.

9. A method in accordance with claim 7, in which the aluminium-bas core material is an alloy of the Aluminum Association 3XXX series.

10. A method in accordance with any of claims 7, 8 and 9, in which the aluminium-base brazing alloy is a silicon-containing aluminium alloy.

11. A method in accordance with any of claims 7, 8 and 9, in which the aluminium-base brazing alloy is an alloy of the Aluminum Association 4XXX series.

12. A method in accordance with any of claims 7 to 11, in which step (a) is performed by vacuum brazing.

13. A method in accordance with any of claims 7 to 12, in which the temperature in step (b) is in the range from 450° to 650°F (233° to 345°C).

14. A method in accordance with any of claims 7 to 13, in which the heating in step (b) is continued for at least 2 hours.

15. A method in accordance with any of claims 7 to 14, further comprising cooling the product of step (a) prior to step (b) to an intermediate temperature below the temperature of step (b) and less than about 550°F (290°C).

16. A method in accordance with claim 15, in which the product of step (a) is cooled to an intermediate temperature less than about 350°F (175°C).

17. A brazed aluminium product formed by:

(a) joining a first aluminium-base material comprising an aluminium-base core material clad with an aluminium-base brazing alloy to a second aluminium-base material by brazing; and

(b) heating the product of step (a) to a temperature in the range from 300° to 800°F (150° to 425°C) for at least 25 minutes.

18. A brazed aluminium product in accordance with claim 17, in which the aluminium-base core material is a non-heat-treatable aluminium-base alloy.

19. A brazed aluminium product in accordance with claim 17, in which the aluminium-base core material is an alloy of the Aluminum Association 3XXX series.

20. A brazed aluminium product in accordance with any of claims 17, 18 and 19, in which the aluminium-base brazing alloy is a silicon-containing aluminium alloy.

21. A brazed aluminium product in accordance with any of claims 17, 18 and 19, in which the aluminium-base brazing alloy is an alloy of the Aluminum Association 4XXX series.

22. A brazed aluminium product in accordance with any of claims 17 to 21, in which step (a) has been performed by vacuum brazing.

23. A brazed aluminium product in accordance with any of claims 17 to 22, in which step (b) has been performed at a temperature in the range from 450° to 650°F (233° to 345°C).

24. A brazed aluminium product in accordance with any of claims 17 to 23, in which the heating in step (b) has been continued for at least 2 hours.

25. A brazed aluminium product in accordance with any of claims 17 to 24, in which the product of step (a) is cooled prior to step (b) to an intermediate temperature below the temperature of step (b) and less than about 550°F (290°C).

26. A brazed aluminium product in accordance with claim 25, in which the product of step (a) is cooled to an intermediate temperature less than about 350°F (175°C).

European Patent Office

EUROPEAN SEARCH REPORT

Application number

EP 87 30 1803

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.4)
X	US-A-3 859 145 (D.S. McGLASSON et al.) * Whole document * ---	1-26	C 22 F 1/04 B 23 K 35/28
A	FR-A-2 185 946 (S.A. DES USINES CHAUSSONS) * Claim 1 * ---	1-26	
A	US-A-2 837 450 (D.C. MOORE et al.) * Claims 1-7; column 2, lines 50-55 * ---	1	
D,A	US-A-2 117 106 (H.F. SILLIMAN) -----		

TECHNICAL FIELDS SEARCHED (Int. Cl.4)

B 23 K
C 22 F

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	08-07-1987	GREGG N.R.

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alon
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